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AN IMAGE INTENSIFIER FOR ELECTRON MICROSCOPY OF POLYMERS.(U)
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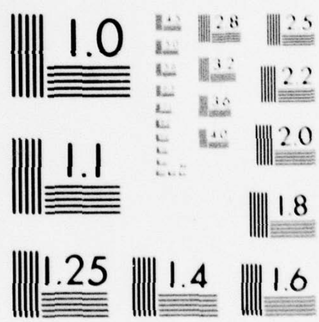

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AN IMAGE INTENSIFIER FOR ELECTRON MICROSCOPY OF POLYMERS.

by

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A unique manipulator for a channel electron multiplier array (CEMA) has been designed and constructed. It allows for easy installation into the viewing chamber of a transmission electron microscope. This device, through a series of motions, positions the CEMA normal to the electron beam, thus amplifying the electron flux while preserving its image. This increase in image brightness permits operation of the microscope with a greatly reduced flux of electrons passing through the sample, thereby — over

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decreasing radiation damage while simultaneously extending the time available for examination of the specimen. The manipulator can then move the CEMA to a remote area of the chamber, thus returning the microscope to its normal operating mode.

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"AN IMAGE INTENSIFIER FOR ELECTRON MICROSCOPY OF POLYMERS"

1. INTRODUCTION

Radiation damage is a problem that severely limits the observation of polymer samples in the electron microscope. While beam damage cannot be completely eliminated, the rate of damage can be slowed, allowing some time for viewing and photographing.

1.1 Radiation Damage in Polymers

High energy electrons in the beam of a transmission electron microscope are known (Vesely, et al, 1976) to cause degradation of crystal structures in hydrocarbons. The interaction of such beam with specimens causes transfer of energy to the macromolecular chains through many inelastic processes. Some bonds break and, depending on the material, thereby create ions, radicals, crosslinks, or broken chains. In polyethylene, alkyl radicals ($\sim CH_2-\dot{C}H-CH_2\sim$) are formed, and hydrogen gas is generated, resulting in the loss of mass. Adjacent radicals then combine to form a crosslink which strains the crystal and distorts the lattice, decreasing the degree of order (Thomas, 1976). Polyoxymethylene and higher polyolefins tend to decay by chain scission (Grubb and Dlugosz, 1976) and, as a consequence, form volatile monomer (Thomas, et al., 1970). These energy transfers give rise to a temperature increase in the irradiated area. This local heating promotes the escape of some low molecular weight material produced by chain scission, and there is, as a result, an increase of hydrocarbon concentration inside the microscope, especially near the sample. The amount by which the vacuum is spoiled depends on the gas evolution rate and the pumping speed of the vacuum system (Vesely, _et al._, 1976). Hydrocarbon gasses in the vicinity of the specimen increase the deposition rate of a surface contamination layer on it.

1.2 Limitations on Electron Microscopy

All these radiation effects severely limit the length of time the relatively undamaged sample can be viewed and photographed. The extent of radiation damage is revealed by an intensity decrease in diffraction maxima or a contrast decrease in the darkfield image. The "crystal lifetime" of the polymer is determined as the time elapsed during which the diffraction pattern degrades into a diffuse halo (Thomas and Ast, 1974). From Figure 1, it is seen that this time occurs when a plateau is reached after an initial exponential decay of intensity (Grubb and Groves, 1971). However, much of the intensity before the plateau is from electrons passing through an already damaged crystal. To account for this, it has been determined that an unacceptable amount of artifact diffraction occurs after 60% of the "crystal lifetime" (Thomas and Ast, 1974). This additional limitation reduces even further the already short time available for observation.

1.3 Techniques for Reducing Radiation Damage

An often-used technique for obtaining electron micrographs from radiation damage-sensitive specimens is the "shooting blind" method. In this technique, one region of the sample is used for focussing (and consequently destroyed), while a distant area is actually used to make the photographic recording (White, 1975). This is advantageous in that no special equipment is needed. However, there are serious drawbacks associated with this method. Any change in specimen height will alter the focus producing unusable micrographs. Also, since the region photographed is not observed beforehand, an uninteresting image might be recorded.

An increase in accelerating voltage can reduce radiation damage by decreasing the ionization rate (Thomas, et al., 1970, Grubb and Groves, 1971). But the quantum detection efficiency (QDE) of the electron microscope plates decreases by almost a factor of 3 when the voltage is increased from 100 kV to 1000 kV, a factor identical to the decrease in damage rate (Thomas, et al., 1970). Therefore, one-third additional electrons are needed to maintain photographic quality. These extra electrons come at the expense of increased damage to the specimen. Until the efficiency of the film emulsion and phosphor screens are improved, there is little advantage to using a high voltage when examining thin polymer films (Thomas, et al., 1970).

Since the ability of the radiation to produce free radicals and crosslinks is linked to kinetics and thermal energy, it is reasonable to expect that cooling the specimen should reduce the damage rate. However, this advantage is diminished when dealing with polymers that decay by chain scission such as polyoxymethylene (Grubb and Groves, 1971). Practical problems related to the microscope itself interfere with this strategy, also.

All the previous techniques require a high electron flux through the sample in order for observation, but what is needed is a device that reduces the amount of electrons damaging the polymer while maintaining an image bright enough for viewing. Such a device would increase the polymer diffracting lifetime, thereby allowing for examination, focussing, and recording of an image with a flux of electrons that would ordinarily be insufficient to detect with a phosphor screen.

2. IMAGE INTENSIFICATION

2.1 Channel Electron Multiplier (CEM)

The channel electron multiplier utilizes the secondary electron emission phenomenon to achieve electron amplification. This device is a highly resistive glass cylinder, internally coated with a secondary electron emitting semiconductor. A uniform axial electrostatic field is created within the channel by applying a potential (typically 1000 volts) across both ends. When an electron enters the channel, it collides with the wall generating secondary electrons which are then accelerated down the tube by the field. These eventually hit the wall causing an avalanche of electrons emerging from the output end (Figure 2). These can then be observed electronically with a collector or optically by placing a phosphor screen near the output.

2.2 Problems with the CEM

When the gain exceeds 10^5, the effects of ion feedback become important. A multiplied electron flux at the output may produce positively charged ions (depending on the residual gas pressure) which are then accelerated toward the input by the field. There they collide with the wall, generating more electrons, and they, in turn, are multiplied as they continue through the channel toward the output. This results in a strong output pulse followed by a series of smaller pulses decreasing in magnitude. To overcome this effect, the channel can be curved, preventing the ions from acquiring enough energy to produce secondary electrons by limiting the distance they travel before hitting the wall (Eschard and Manley, 1971).

The maximum gain achievable (approximately 10^8) is limited by an effect due to space charge, which is the electrostatic charge of the electron cloud itself. As the electrons approach the output, the density of electrons in the cloud increases. This causes a self-repulsion, driving the electrons to the wall before they have acquired sufficient energy to create secondary electrons. Therefore, the electron flux reaches a self-limiting saturation level.

2.3 Channel Electron Multiplier Array (CEMA)

The CEMA can be created by fusing millions of these short (~1 mm), 25 μ diameter channels together, parallel to each other. This produces a plate that allows two-dimensional information to be amplified. By mounting a phosphor screen (that is itself deposited on a fiber optic substrate) just below the channel output, the incident electron flux can be visualized optically. After proper biasing, the CEMA would produce an intensified image through electron multiplication. When placed in the viewing chamber of a transmission electron microscope, it is possible to reduce the electron flux through the sample, even while gaining an increase in image brightness. As the electron flux incident on the sample is decreased, there is a corresponding decrease in radiation damage to the sample. Since the radiation damage rate is very low, there is now more time available for focussing and photographing before the "60% crystal lifetime" (Thomas and Ast, 1974) is reached.

2.4 Operation of the Image Intensifier

A problem remains in where to locate the channelplate. A previously employed method involves placing the intensifier beneath the camera chamber (Thomas and Ast, 1973). This position allows for normal operation of the microscope, but an externally mounted periscope arrangement must be used to sight up the column and see the phosphor screen of the intensifier. A more compact arrangement would place the CEMA in the space just above the regular phosphor screen of the microscope. A front-silvered mirror is suspended below the intensifier at the proper angle and thereby allows the output to be seen through the normal viewing window. Photographs can also be easily taken through this same window with a 35 mm camera externally mounted on a tripod. Of course, the resolution of these photographs is limited by the channel diameter and the percent of open area in the plate (55%). The best means of recording any image is by using the existing camera system in the electron microscope. This was accomplished by constructing a unique manipulator that can both support the channelplate perpendicular to the beam when it is to be used for focussing and then to rotate the CEMA into a vertical position at the rear of the viewing chamber (Figure 3). The manipulator can move the CEMA into this out-of-the-way position, and then the standard photographic system of the microscope itself can be used to make maximum resolution recordings directly from the electron beam itself. This mobility of the CEMA allows an unobstructed view of nearly the entire phosphor screen so that normal operation of the microscope and camera can be accomplished. Since the electron beam is at a very low intensity, micrographs are simply exposed for a sufficiently long period of time to acquire the necessary number of electrons to record a quality image.

2.5 Design of the Manipulator

The manipulator is designed so that it can be easily installed and removed through the front window of a JEOL 100B electron microscope. It is attached to the phosphor screen of the microscope with 4 screws at the rear of the chamber. A mechanical rotary feedthrough, replacing an unused rear window, connects to the manipulator via a flexible shaft. This is geared to a vertical screw that drives the rear of the channelplate up and down. The front of the plate is attached in two places to a pivoting arm that allows the front to swing forward while the back is raised. The arm motion releases the front end of a mirror allowing it to slide down and lodge at an angle designed to permit observation of the intensifier phosphor screen (output). Reversing the direction of the feedthrough, lowers the back of the plate and swings the arm nearly vertical, causing the mirror to slide up and lie flat against the fiber optic substrate. The rotation can be performed either manually with a geared knob or with a motor. (In the work reported here, a motor was mounted outside the microscope and performed the position change in only 8 seconds. Travel up and down was limited by microswitches and adjusting screws which immediately turned off the motor, stopping the manipulator). The space limitation in the viewing chamber necessitated the minimization of size, motion and the height of the intensifier above the phosphor screen. Because of these basic design considerations, the image intensifier and manipulator can be easily adapted to fit most microscopes.

2.6 Significance of Image Intensifier

As discussed earlier, the advantage in using an intensifier is due to the decreased radiation damage during focussing. During the minute it takes to locate and focus an interesting area, the sample receives enough electrons/cm^2 to record 9 bright field micrographs (Thomas and Ast, 1974). By using the CEMA for focussing, not only the number of images recorded increases but the quality of them is higher because the sample receives less damage during each exposure.

There are other intensification systems available commercially that work as well as the channelplate system, but their cost is very much higher (>$30,000 as opposed to $4,000). Therefore, unless electronic processing of the image is needed, the more direct and inexpensive channelplate should be used.

While this system is needed to reduce radiation damage in thin polymer samples, it can also be used for morphological studies in thicker samples. Even microscopy on metal samples can be improved and made easier by allowing thicker samples to be observed.

ACKNOWLEDGEMENTS

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Figure Captions

Figure 1

Total intensity of diffracted electrons, J, for polyethylene crystal. The values shown correspond to an incident flux through samples 4×10^{-5} A/cm^2. (Thomas and Ast 1974).

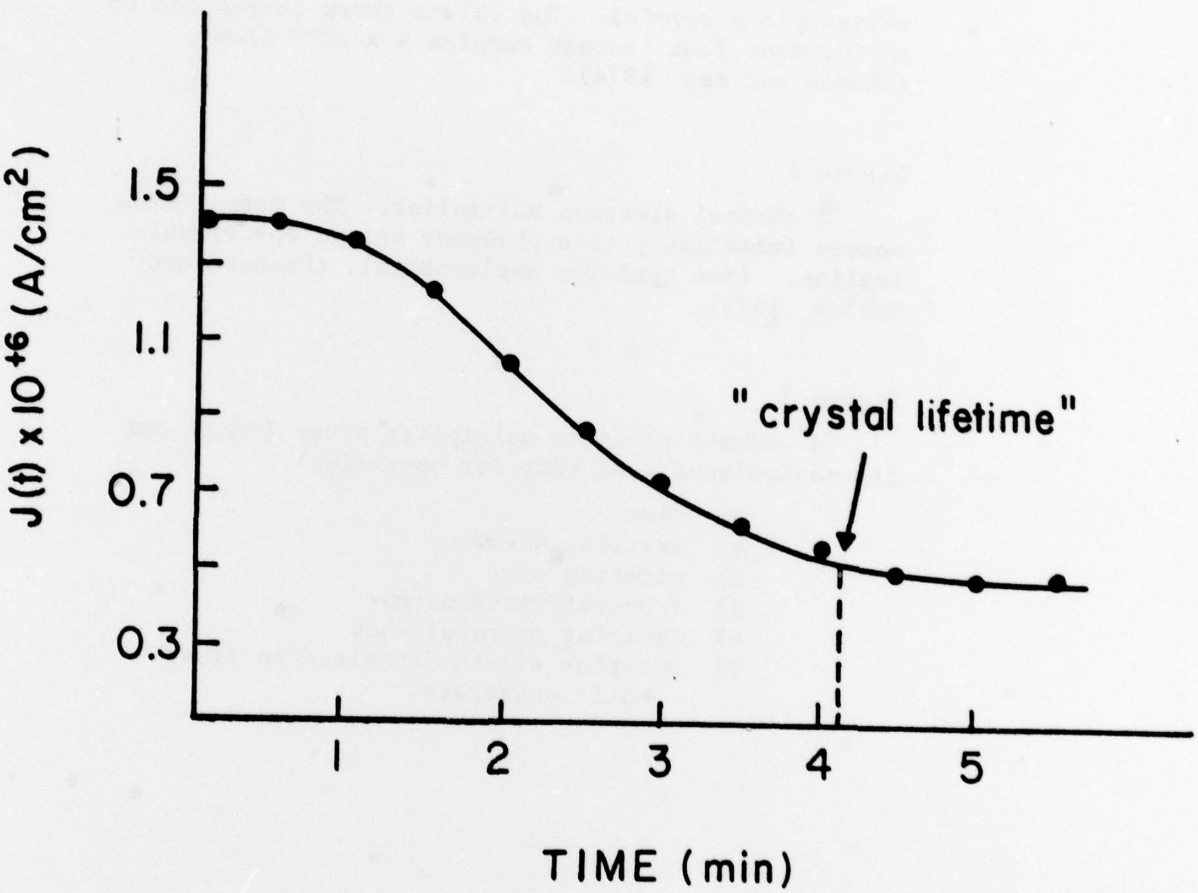
Figure 2

A channel electron multiplier. The output flux passes immediately to a phosphor screen for visualization. (See text for explanation). (Eschard and Manley 1971).

Figure 3

A channel electron multiplier array (CEMA) and its manipulator (see text for operation).

a) CEMA
b) vertical screw
c) pivoting arm
d) front-silvered mirror
e) limiting microswitches
f) phosphor screen deposited on fiber optic substrate



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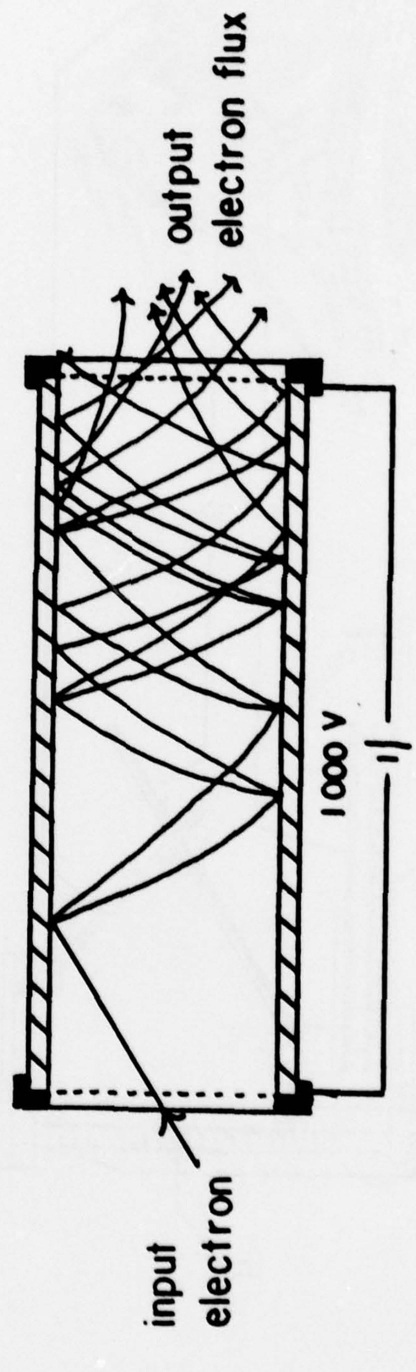

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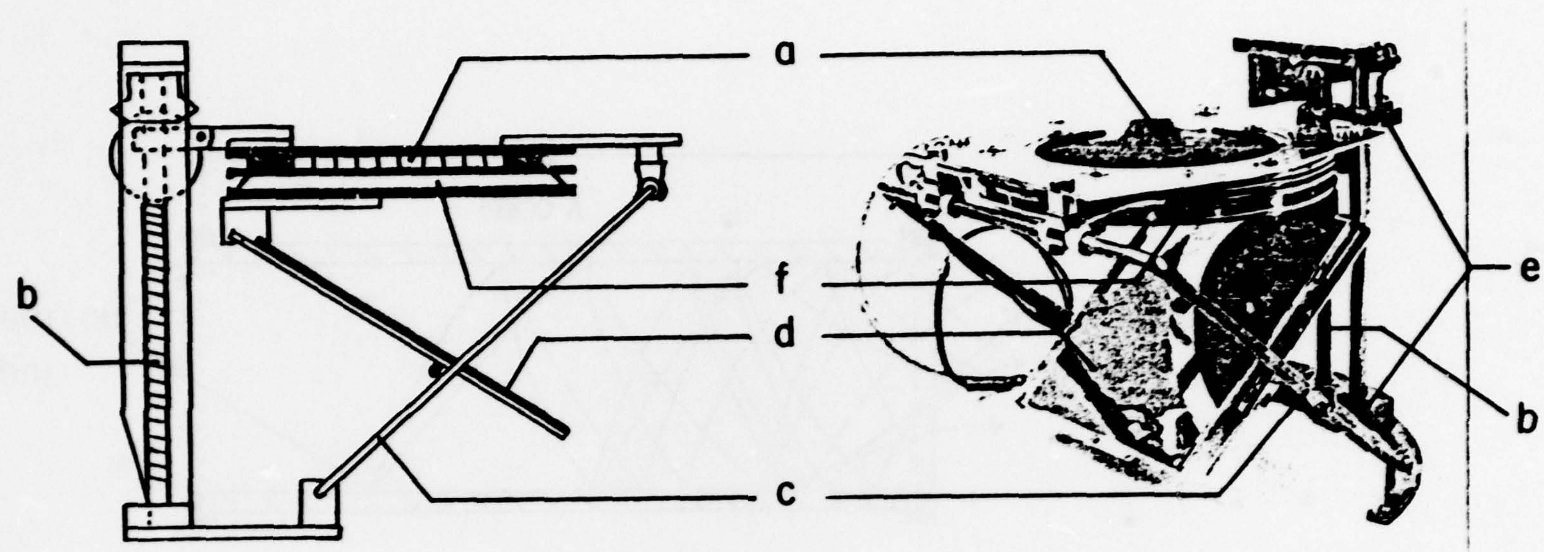

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